





# TECHNICAL REPORT RDMR-WD-16-62

# TERAHERTZ (THZ) OPTICAL PARAMETERS OF THREE-DIMENSIONAL (3-D) PRINTING MATERIALS

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# I. INTRODUCTION

Three-dimensional (3-D) printing is becoming a popular prototyping technique for industry and the general hobbyist. Little is known about the structural integrity of a printed structure, and its mechanical strength may vary greatly depending on the material utilized, technology for generating the structure, and the printer. Studying the mechanical properties of a 3-D print can be difficult, and employing stress techniques is commonly used in metal and also in both the conductive/nonconductive extrusion industry. Quantifying parts manufactured using additive processes is an unknown, as is understanding the material properties of additively created structures versus those created using standard processes, such as injection molding, casting, or machining. Terahertz (THz) radiation and imaging has become a topic of research as an optical technique to study these materials because THz radiation can penetrate many visibly opaque dielectrics, such as printed materials, and provide imagery through scanning techniques at submillimeter resolution. Before a 3-D printed structure can be imaged quantitatively, the optical properties of these materials at THz frequencies [1, 2] must be known. The following reports on measurements to calculate the absorption coefficients of three plastic materials commonly used in 3-D printing. The following are three general interactions that THz radiation can have with homogenous bulk materials:

- Reflection (R)
- Transmission (T)
- Absorption (A)

A THz ellipsometer was used to measure the transmission of several 3-D printed slabs with known thicknesses. A Vector Network Analyzer (VNA) was also used to measures the reflection of THz radiation from the samples. With both the reflection and transmission measurements, the materials' absorption coefficients can be calculated.

# II. THEORY AND EXPERIMENTAL SETUP

Beer's law was used to calculate the absorption coefficient,  $\alpha$ , based on the thickness,  $\ell$ ; R; and T of the sample. Assuming homogeneity in the sample composition the absorption coefficient is expressed according to Beer's law as the following:

$$\alpha = -\ln \left( T / \left( 1 - R \right) \right) / \ell. \tag{1}$$

The values for Transmission, T, are frequency dependent. Therefore, the Absorption, A, coefficient is also frequency dependent. For this experiment, Reflection, R, is assumed to be constant across all frequencies [1]. Equation 1 then becomes a function of frequency:

$$\alpha(f) = -\ln \left( T(f) / (1 - R) \right) / \ell \tag{2}$$

Three materials, each tinted with two different colors, were measured. The materials were High Impact polystyrene (HIPS), acrylonitrile butadiene styrene (ABS), and polylactic acid (PLA). All three materials are common, low-cost, 3-D printed materials. Pictures of the HIPS samples are shown in Figure 1. Samples of the other two materials were similar in size (approximately 5-by-5 centimeters (cm) wide and 2 millimeters (mm) thick). The test articles

were created using a Lulzbot TAZ Mini and printed with 100 percent (%) infill. This test could be expanded to look at the varying infill percentages (for example, bulk density) that can be created using additive processes.



Figure 1. Representative Printed Slab Samples (HIPS)

The transmission measurements were performed on a J. A. Woollam Variable Angle Spectroscopic Ellipsometer (VASE) over four frequency bands: 220 to 330; 330 to 500; 650 to 1,000; and 1,000 to 1,500 gigahertz (GHz). A picture of the ellipsometer is shown in Figure 2. Note that two white circular objects represent the polarization generator on the left and the analyzer on the right. The plastic samples were placed in the beam path at normal incidence, and the spectral transmittance normalized to a transmission plot without a sample were measured. Figure 3 shows the orange HIPS sample mounted for measurements. Baseline scans without the sample and calibration alignments of the ellipsometer were performed before each frequency scan.

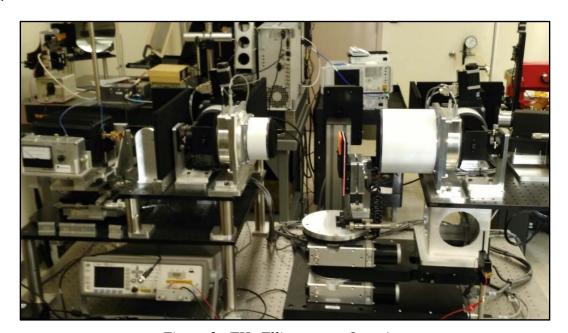


Figure 2. THz Ellipsometer Overview

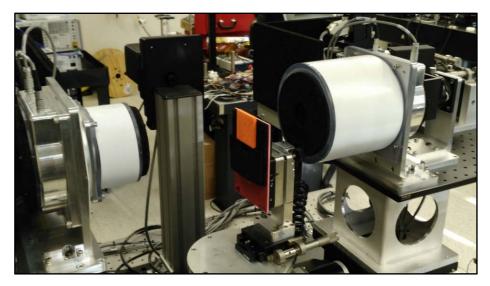


Figure 3. HIPS Sample Mounted for Transmission Measurements

The ellipsometer does not support normal incidence reflectance measurements. Instead, reflectance was measured with an Agilent N522A PNA, a WR 2.2 325-500 GHz transceiver, and a WR 2.2 325-500 GHz receiver [3]. The THz units are network extenders by Virginia Diodes, Incorporated, which are integrated into a 26.5 GHz network analyzer. Custom mechanical calibration samples were used to calibrate the VNA over 325 to 500 GHz for the S<sub>11</sub> parameter measurements. A concave mirror, as shown in the bottom right-hand side of Figure 4, was placed at its focal distance from the transceiver to collimate the THz beam. A biconvex lens was placed further down the collimated beam path to focus the beam on the sample, ensuring that all THz reflection reflects off or passes through the sample under test, as shown in Figure 5. Figure 5 demonstrates how aluminum foil serves as an aperture to limit the beam size to the size of the lens. A large mirror was placed directly behind the sample, which is assumed to have a reflectance of 1 (perfect mirror) for normalization purposes.

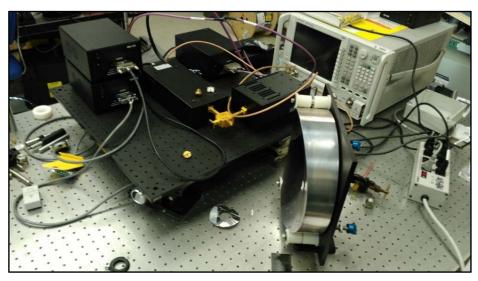


Figure 4. Normal Incidence Reflectance Measurements Showing Transceiver, Receiver, VNA, and Collimating Mirror

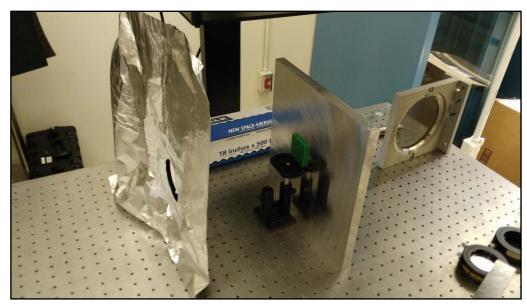


Figure 5. Green Sample Shown in Reflection Geometry

In Figure 5, a green sample was placed in front of the mirror and at the focal length of the THz lens. The  $S_{11}$  measurement on the VNA, which is equivalent to a reflection measurement, shows the reflected amplitude due to all surfaces in the system. By sweeping the THz frequency and performing a Fast Fourier Transform (FFT), the reflections can be separated in time, as shown in Figure 6. Figure 6 shows the reflected amplitude due to the mirror only, which occurs at approximately 6.5 nanoseconds (ns) and has a strength of approximately -10 decibels (dB). This peak response was equivalent to a reflection of 1 and used for normalization.

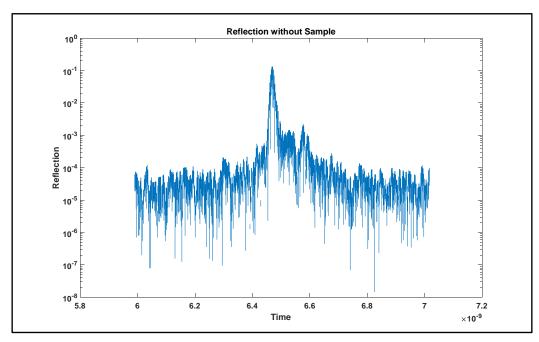


Figure 6. Reflection From Mirror and Without Sample

Figure 7 shows the same measurement but with the green sample placed against the front of the mirror. Note the two reflections, one due to the front of the green sample and the other due to the mirror after the THz beam passed through the sample. The green sample reflection (first peak) is at approximately -18 dB (or -8 dB) compared to perfect reflection. The second reflection due to the mirror is significantly reduced due to the absorption of THz radiation as it has to pass through the sample twice before reaching the detector. Hence, the transmission can be measured as well. However, the ellipsometer is slightly more convenient and performs better at reducing standing waves, which could lead to false measurements. The reflectance of HIPS, ABS, and PLA from 300 to 500 GHz were as follows: HIPS = 13%, ABS = 23%, PLA = 14%.

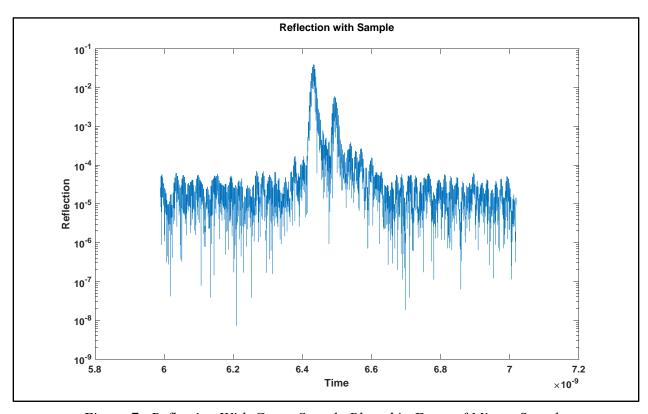


Figure 7. Reflection With Green Sample Placed in Front of Mirror Sample

# III. RESULTS AND DISCUSSION

As noted previously, the transmission of the plastic samples were measured using an ellipsometer. The results of several AT p-polarized (pp) and s-polarized (ss) THz transmission scans are shown in Figures 8 through 10. As expected, the transmission decreased with increasing frequency for all printing materials, and color tint has no effect on the optical properties at THz frequencies. HIPS is the most transparent materials with nearly 90% transmittance at 220 GHz and 30% transmittance at 1,000 GHz. PLA and ABS become essentially opaque above 650 GHz using the ellipsometer, suggesting a transmission of less than 0.001 or 30 dB which is the sensitivity limit of the ellipsometer. The resonant pattern is due to standing waves forming inside the samples.

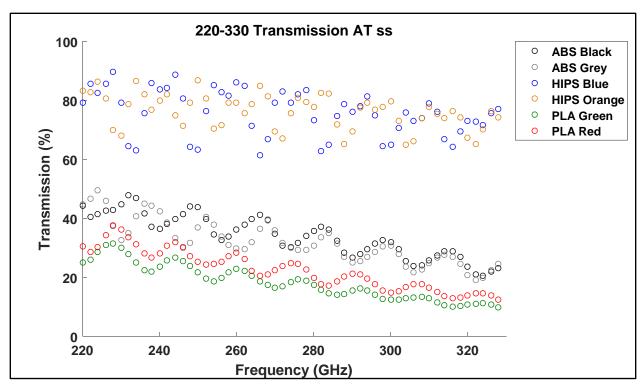


Figure 8. Transmission at 220 to 330 GHz

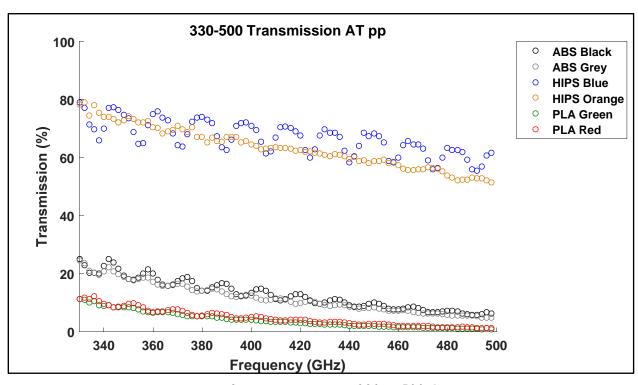


Figure 9. Transmission at 330 to 500 GHz

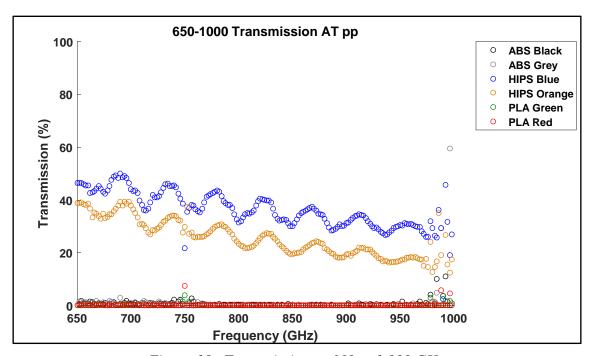


Figure 10. Transmission at 660 to 1,000 GHz

From the aforementioned reflectance measurements and the transmission measurements, the absorption coefficients were calculated and plotted in Figures 11 through 13. The absorption coefficient of HIPS ranged from 0.5 to 3 cm<sup>-1</sup> for HIPS. Figure 13 confirms that the absorption coefficients of PLA and ABS are too large to be measured at 660 to 1,000 GHz using the ellipsometer.

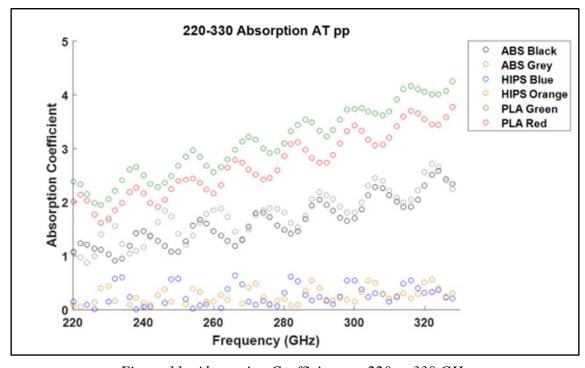


Figure 11. Absorption Coefficients at 220 to 330 GHz

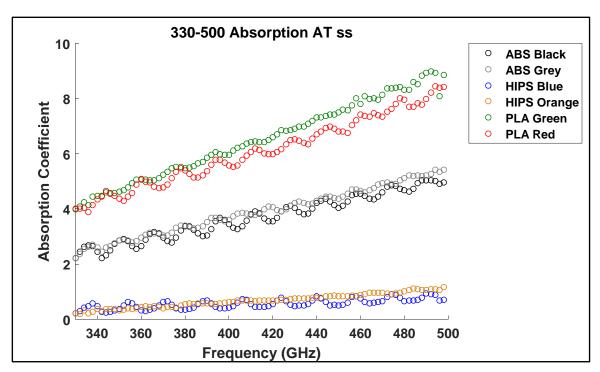


Figure 12. Absorption Coefficients at 330 to 500 GHz

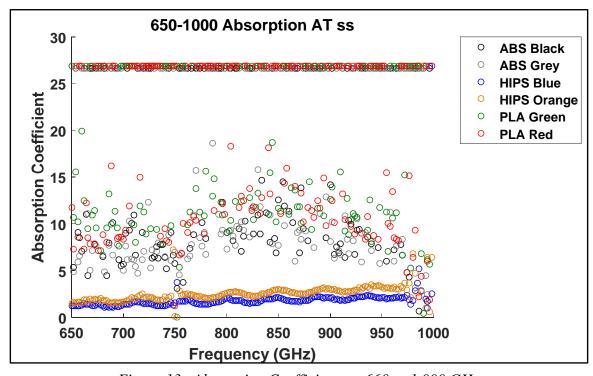


Figure 13. Absorption Coefficients at 660 to 1,000 GHz

For all of the plastic samples (ABS, HIPS, and PLA), the absorption coefficient (cm<sup>-1</sup>) increases linearly with frequency. The plastics in order from least to greatest absorption coefficients are as follows: HIPS < ABS < PLA.

# IV. CONCLUSION

The results from these tests show that the absorption coefficient increases linearly for all plastic samples. HIPS is the most transparent at THz frequencies, followed by ABS and PLA. Nevertheless, using a heterodyne technique rather than square law detection, either material allows THz radiation to penetrate, suggesting that nondestructive imaging methods are a valid quality control tools at THz frequencies. Several THz imaging methods will be investigated in the future.

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# LIST OF ABBREVIATIONS, ACRONYMS, AND SYMBOLS

% percent < less than

3-D Three-Dimensional

A Absorption

ABS acrylonitrile butadiene styrene

cm centimeter dB decibel

FFT Fast Fourier Transform

GHz gigahertz

HIPS High Impact polystyrene

mm millimeters
ns nanosecond
PLA polylactic acid
pp p-polarized
R Reflection
ss s-polarized

T Transmission

THz terahertz

VASE Variable Angle Spectroscopic Ellipsometer

VNA Vector Network Analyzer